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PREPARATION AND PROPERTIES OF THE SYSTEM CR2-KRNO03(2
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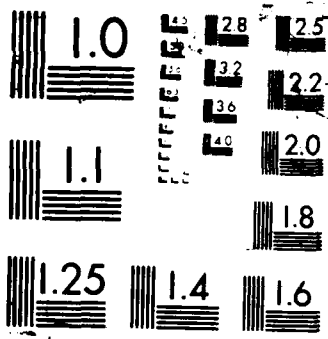
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TECHNICAL REPORT NO. 13

Preparation and Properties of the System $\text{Cr}_{2-x}\text{Rh}_x\text{O}_3$ ($2 \geq x \geq 0$)

by

Y-C. Zhang, R. Kershaw, K. Dwight and A. Wold

Prepared for Publication in
JOURNAL OF THE LESS-COMMON METALS

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PREPARATION AND PROPERTIES OF THE SYSTEM $\text{Cr}_{2-x}\text{Rh}_x\text{O}_3$ ($2 \geq x \geq 0$)

by

Y. C. Zhang, R. Kershaw, K. Dwight and A. Wold

Chemistry Department, Brown University, Providence, RI 02912

ABSTRACT

Members of the system $\text{Cr}_{2-x}\text{Rh}_x\text{O}_3$ ($2 \geq x \geq 0$) were prepared by thermal decomposition of the nitrates. Magnetic measurements indicate that the chromium-rich members are antiferromagnetic and the spin-only moment, extrapolated from the high temperature portion of the susceptibility curves, corresponds to Cr(III). Reduction studies indicate that the rhodium was stabilized in the solid solution and that at elevated temperatures between 1100 and 1200°C the mixed oxides were reduced to either the elements or a rhodium-chromium alloy.

INTRODUCTION

Khanolkar [1] reported that rhodium metal and chromium(III) oxide react to form an ilmenite phase CrRhO_3 with $a = 5.42 \text{ \AA}$ and $\alpha = 55^\circ 12'$.

Shaplygin et al. [2] showed from DTA and x-ray studies that below 1210°, Cr_2O_3 and CrRhO_3 coexist at 0-50 mole percent Rh_2O_3 and below 1060° CrRhO_3 and Rh_2O_3 coexist at 50-100 mole percent Rh_2O_3 . The samples were prepared by heating mixtures of Cr_2O_3 and Rh_2O_3 at 900-1000°C for 150-200 hours.

There appears to be little else published concerning this system. Since both end members crystallize with the corundum structure [3, 4], with cell parameters of $a = 4.94$ and 5.13 \AA , and $c = 13.38$ and 13.85 \AA for Cr_2O_3 and Rh_2O_3 , respectively, the reexamination of the earlier work using better synthetic methods was deemed advisable.

Furthermore, it was reported by Striter and Mantell [5] that hydrogen reduction of Cr_2O_3 to chromium was found to be feasible above 1130°C at very low water concentrations. However, the presence of a small quantity of residual water vapor retarded the rate of reduction. This was confirmed by Hussein et al. [6] who succeeded in reducing Cr_2O_3 with hydrogen to Cr at 1200°C . The authors also reported [7] that the addition of metallic iron increases markedly the extent of reduction. In this study, reduction studies of members of the system $\text{Cr}_{2-x}\text{Rh}_x\text{O}_3$ were carried out as a function of increased concentration of the rhodium to see if the results were similar to those found for the $\text{Cr}_{2-x}\text{Fe}_x\text{O}_3$ system.

EXPERIMENTAL

Samples of $\text{Cr}_{2-x}\text{Rh}_x\text{O}_3$ ($2-x>0$) were prepared by codecomposition of rhodium nitrate and chromium nitrate. The calculated weights of $\text{Cr}(\text{NO}_3)_3 \cdot 2\text{H}_2\text{O}$ and $\text{Rh}(\text{NO}_3)_3 \cdot 2\text{H}_2\text{O}$ were dissolved in water and the solution was dried at 150°C for 12 hours, ground, and decomposed at 400°C for 24 hours. The sample was then ground and heated at 1000°C for 48 hours. Rhodium metal was prepared by reducing Rh_2O_3 with $\text{H}_2/\text{Ar}(15/85)$ at 500°C for 24 hours in a horizontal tube

furnace. The Rh_2O_3 was prepared by decomposing $\text{Rh}(\text{NO}_3)_3 \cdot 2\text{H}_2\text{O}$ at 800°C in air for 20 hours and Cr_2O_3 was prepared by decomposing $\text{Cr}(\text{NO}_3)_3 \cdot 2\text{H}_2\text{O}$ at 900°C in air for 24 hours.

Temperature programmed reduction studies were carried out on four compositions ($x = 0.4, 1, 1.6$ and 2) using a Cahn 113 thermal balance. These compositions were chosen in order to study the stabilization of rhodium(III) towards reduction in the solid solution series $\text{Cr}_{2-x}\text{Rh}_x\text{O}_3$. Each sample ($\sim 30\text{mg}$), after being placed in the balance, was purged with a stream of $\text{H}_2/\text{Ar}(15/85)$ in order to obtain a constant initial weight. The H_2/Ar gas was predried over a P_2O_5 trap and the flow rate was 60cc/min . The temperature was then increased to 1000°C at a rate of $50^\circ/\text{hr}$.

In order to study the complete reduction of Cr(III) , the end member Cr_2O_3 was reduced in a Lindberg furnace at 1200°C for 36 hours in flowing hydrogen with a flow rate of 60cc/min . The hydrogen was predried over a P_2O_5 trap. For compositions with $2 \geq x \geq 1$, the reductions were carried out in a Lindberg furnace at 1100°C . A sample weight of approximately 100 mg was put in a silica crucible. At the completion of the reduction, the furnace was turned off and the sample allowed to cool to room temperature in the furnace.

X-ray powder diffraction patterns of all samples were obtained using a Philips diffractometer and monochromated high intensity $\text{CuK}\alpha_1$ radiation ($\lambda = 1.5405\text{\AA}$). The diffraction patterns were taken in the range of $12^\circ - 2\theta$ to 80° with a scan rate of $1^\circ 2\theta/\text{min}$ and a chart speed of 30in/hr . In order to obtain the cell parameters, slow scan x-ray patterns were obtained with a scan

rate of $0.25^\circ \text{C}/\text{min}$. Cell parameters were obtained from a least squares refinement of the data with the aid of a computer program which corrected for the systematic experimental errors. Magnetic susceptibility of the oxide samples was measured using a Faraday balance [8] from liquid nitrogen temperature to 763 K. A field strength of 10.4 kOe was used in the measurements.

RESULTS AND DISCUSSION

Members of the system $\text{Cr}_{2-x}\text{Rh}_x\text{O}_3$ ($2 \geq x \geq 0$) were prepared by decomposition of the nitrates and cell parameters were measured and are shown as a function of increasing rhodium oxide concentration in Fig. 1. The magnetic susceptibility data are given in Table 1. The moments reported were extrapolated from the high temperature portion of susceptibility curves plotted in Fig. 2. The measured moment corresponds closely to the value $3.9\mu_B$ reported for Cr_2O_3 .

The small observed variation in the observed moments (± 0.1) was due to the limited temperature range of the high temperature measurements. The observed Neel temperatures for the chromium-rich members are consistent with the antiferromagnetic behavior of $3d^3$ chromium. Rhodium does not contribute to the observed moments but does reduce the strength of the antiferromagnetic interactions.

The results of the temperature programmed reduction studies for compositions where $x = .4, 1, 1.6$ and 2 are shown in Fig. 3. The shift in the initial sharp reduction step indicates the increased stabilization of $Rh(4d^6)$ from $90^\circ C$ for pure Rh_2O_3 to $900^\circ C$ for $Cr_{1.6}Rh_{0.4}O_3$. For the compositions $CrRhO_3$ and $Cr_{.4}Rh_{1.6}O_3$, the total weight loss was greater than that calculated for the reduction of $Rh(III)$ to $Rh(0)$. Therefore, it was suspected that some of the chromium(III) was also being reduced. A mechanical mixture of Cr_2O_3 and Rh to give a ratio of Rh/Cr of $2:1$ was reduced in pure hydrogen ($60cc/min$) at $1000^\circ C$ for 24 hours. The product was subjected to x-ray analysis and corresponded to the alloy Rh_2Cr (Fig. 4). From the TPR data shown in Fig. 5, the observed weight loss was found to correspond to that calculated for the complete reduction of Cr_2O_3 . This is similar to the observation of Hussein et al. (7) for the influence of iron on the reduction of Cr_2O_3 .

A series of solid solutions from Rh_2O_3 to $CrRhO_3$ were completely reduced under flowing hydrogen and complete reduction was achieved at $1100^\circ C$ after 36 hours. The results are given in Table 2 and indicate the formation of a pure (ϵ) alloy phase where $Cr > 0.5$. The reduction of Cr_2O_3 to chromium metal, as detected by x-ray analysis, was carried out in a flowing hydrogen atmosphere ($60cc/min$) for 36 hours at $1200^\circ C$. All of the compositions between $CrRhO_3$ and Cr_2O_3 can be completely reduced under these conditions.

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Table 1
MAGNETIC PROPERTIES OF $\text{Cr}_{2-x}\text{Rh}_x\text{O}_3$

Composition	Magnetic Moment of Cr^{3+} (μ_B)	Néel Temperature T_N (K)	Weiss Constant θ (K)
Cr_2O_3	4.0	315	-692
$\text{Cr}_{1.8}\text{Rh}_{0.2}\text{O}_3$	4.0	270	-546
$\text{Cr}_{1.6}\text{Rh}_{0.4}\text{O}_3$	4.0	205	-395
$\text{Cr}_{1.5}\text{Rh}_{0.5}\text{O}_3$	4.0	180	-331
$\text{Cr}_{1.4}\text{Rh}_{0.6}\text{O}_3$	4.0	150	-283
$\text{Cr}_{1.3}\text{Rh}_{0.7}\text{O}_3$	3.8	125	-194
CrRhO_3	3.8	---	-102
$\text{Cr}_{0.4}\text{Rh}_{1.6}\text{O}_3$	3.8	---	- 50

Table 2

X-RAY ANALYSIS OF $\text{Cr}_{2-x}\text{Rh}_x\text{O}_3$ AFTER REDUCTION

Initial Composition	Phases of Reduction Products	Cell Parameters	
		a (Å)	c (Å)
Rh_2O_3	cubic (γ -Rh)	3.803	
$\text{Cr}_{0.4}\text{Rh}_{1.6}\text{O}_3$	cubic (γ) + trace hex. (ϵ)	3.780*	
$\text{Cr}_{0.48}\text{Rh}_{1.52}\text{O}_3$	hex. (ϵ) + trace cubic (γ)	2.685**	4.290
$\text{Cr}_{0.5}\text{Rh}_{1.5}\text{O}_3$	hex. (ϵ)	2.687	4.290
$\text{Cr}_{0.67}\text{Rh}_{1.33}\text{O}_3$	hex. (ϵ)	2.678	4.269
CrRhO_3	hex. (ϵ)	2.665	4.251

* Cell parameter of cubic rhodium phase.

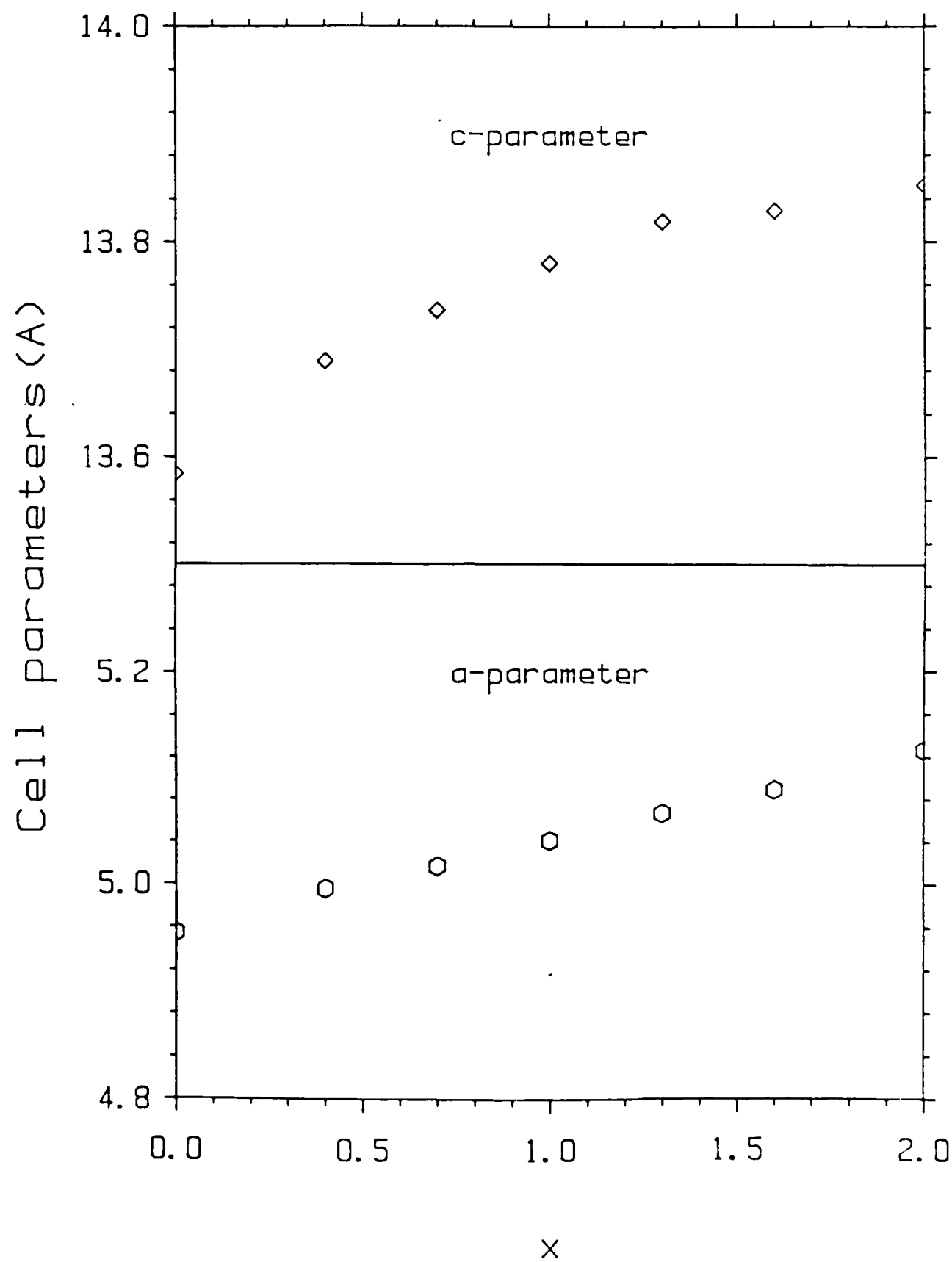
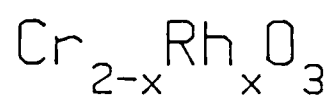
** Cell parameters of hexagonal phase.

Fig. 2. X-ray phase analysis of the mixture of Rh and Cr_2O_3 after 24 hr of heating in flowing H_2 at 1000°C.

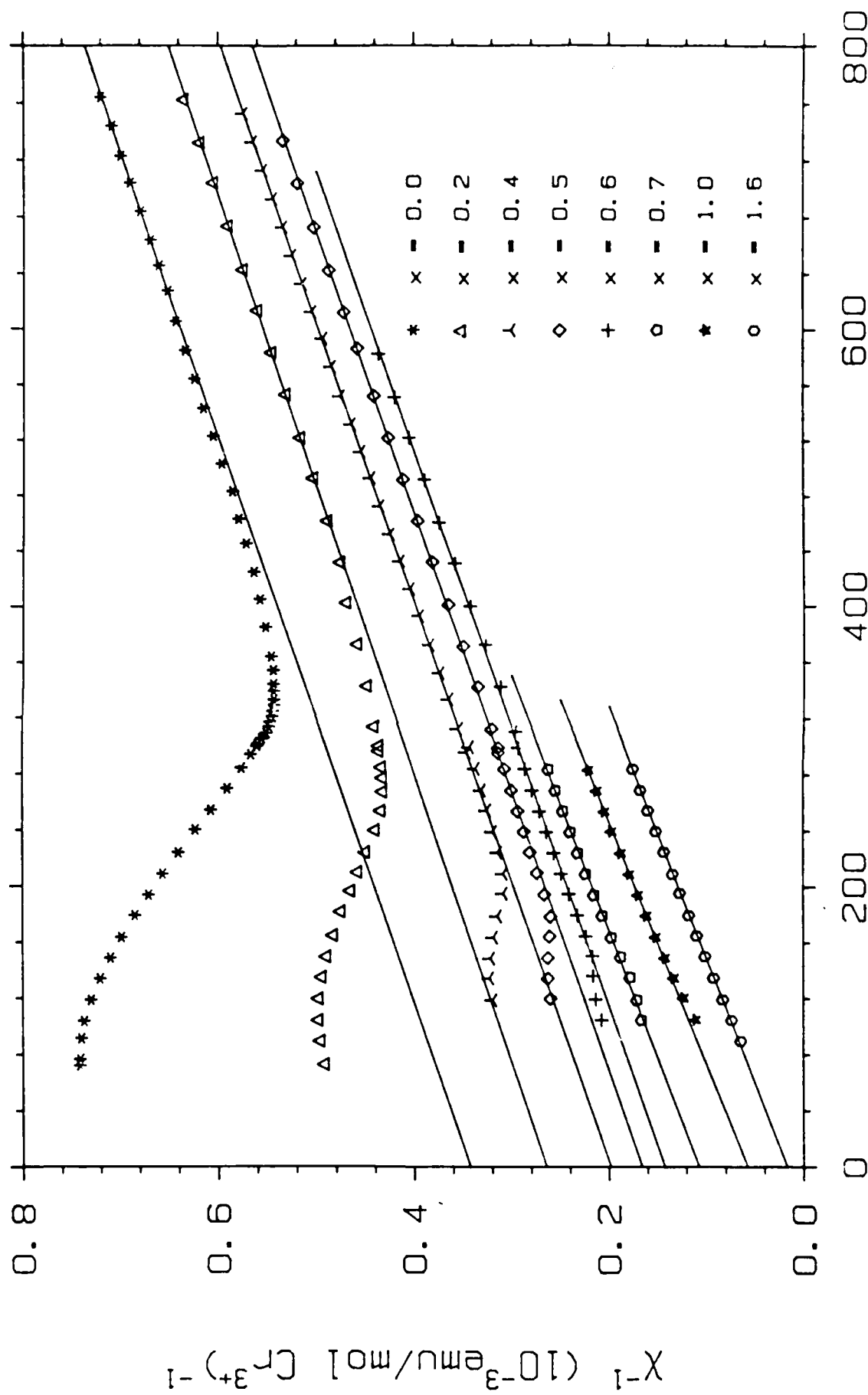
Fig. 3. Relative weight as function of temperature for members of the system $\text{Cr}_2\text{O}_3\text{-Rh}_2\text{O}_3$ heated at 50°C/hr in 60 cc/min of flowing H_2 -Ar 15, 85%.

Fig. 4. X-ray phase analysis of a mixture of Rh and Cr_2O_3 before and after reduction in flowing H_2 for 24 hr at 1000°C.

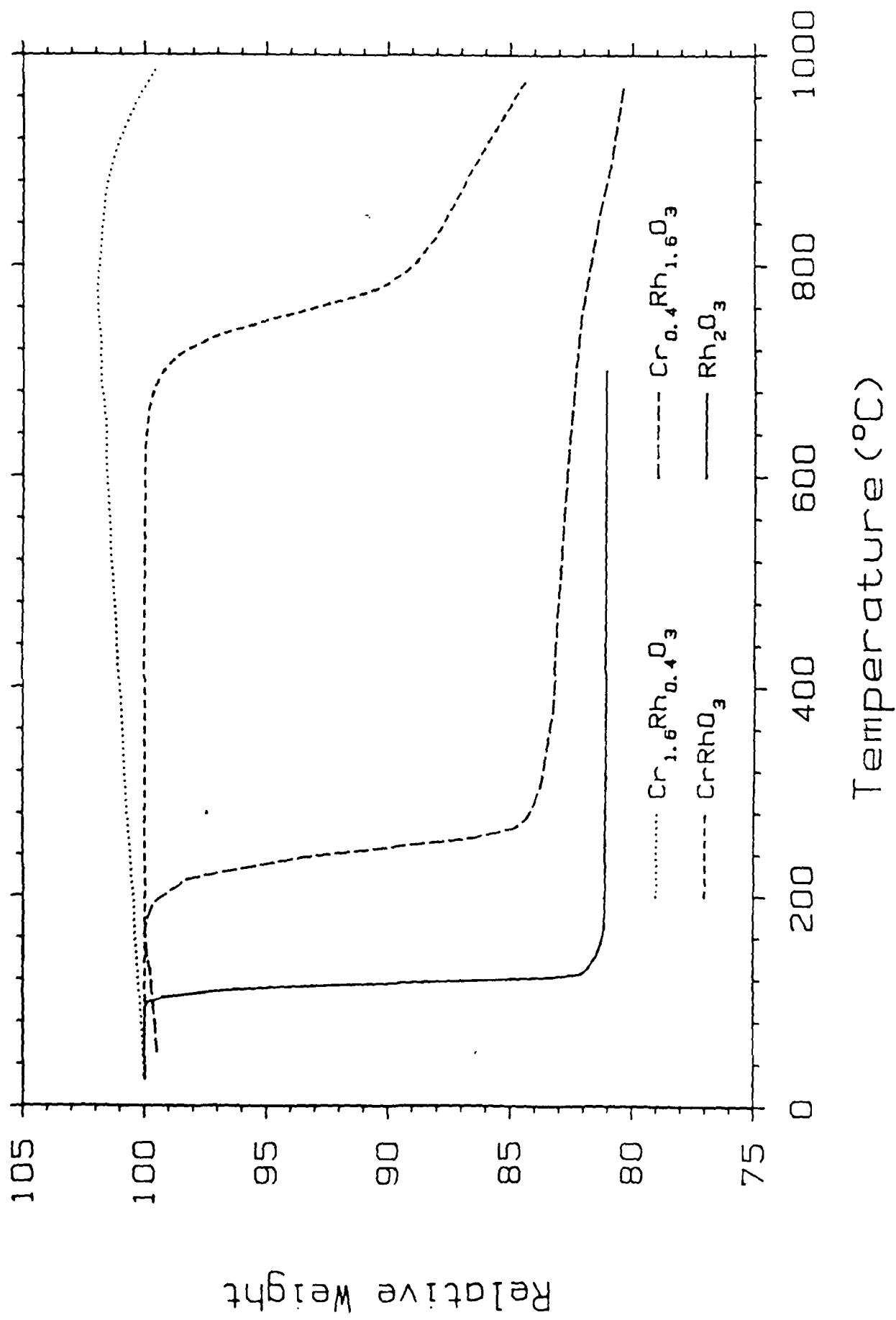
Fig. 5. Time profiles of temperature and corresponding relative weight for a mixture of Rh and Cr_2O_3 heated in flowing H_2 .



$\text{Cr}_{2-x}\text{Rh}_x\text{O}_3$

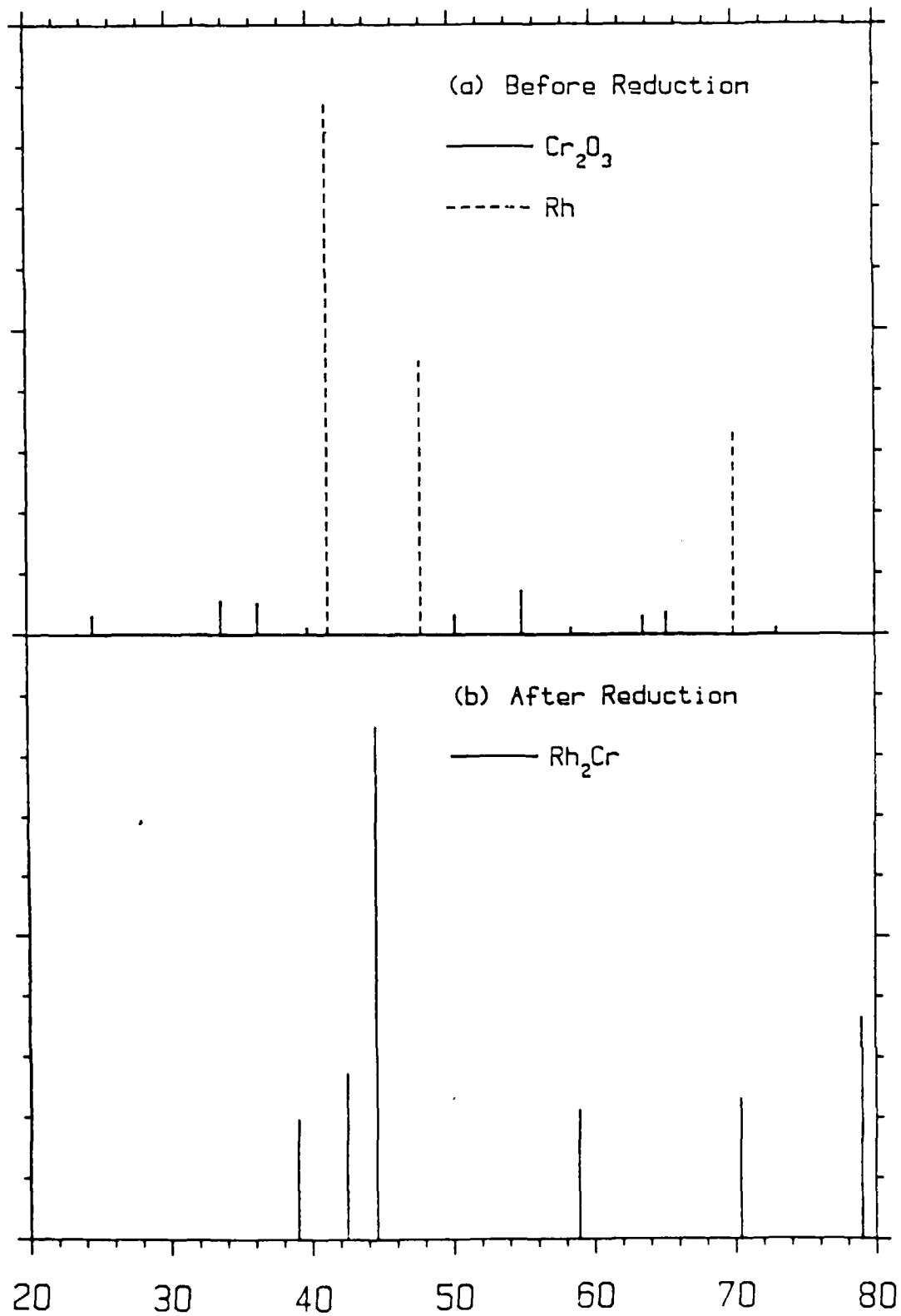


TPR of $\text{Cr}_{2-x}\text{Rh}_x\text{O}_3$

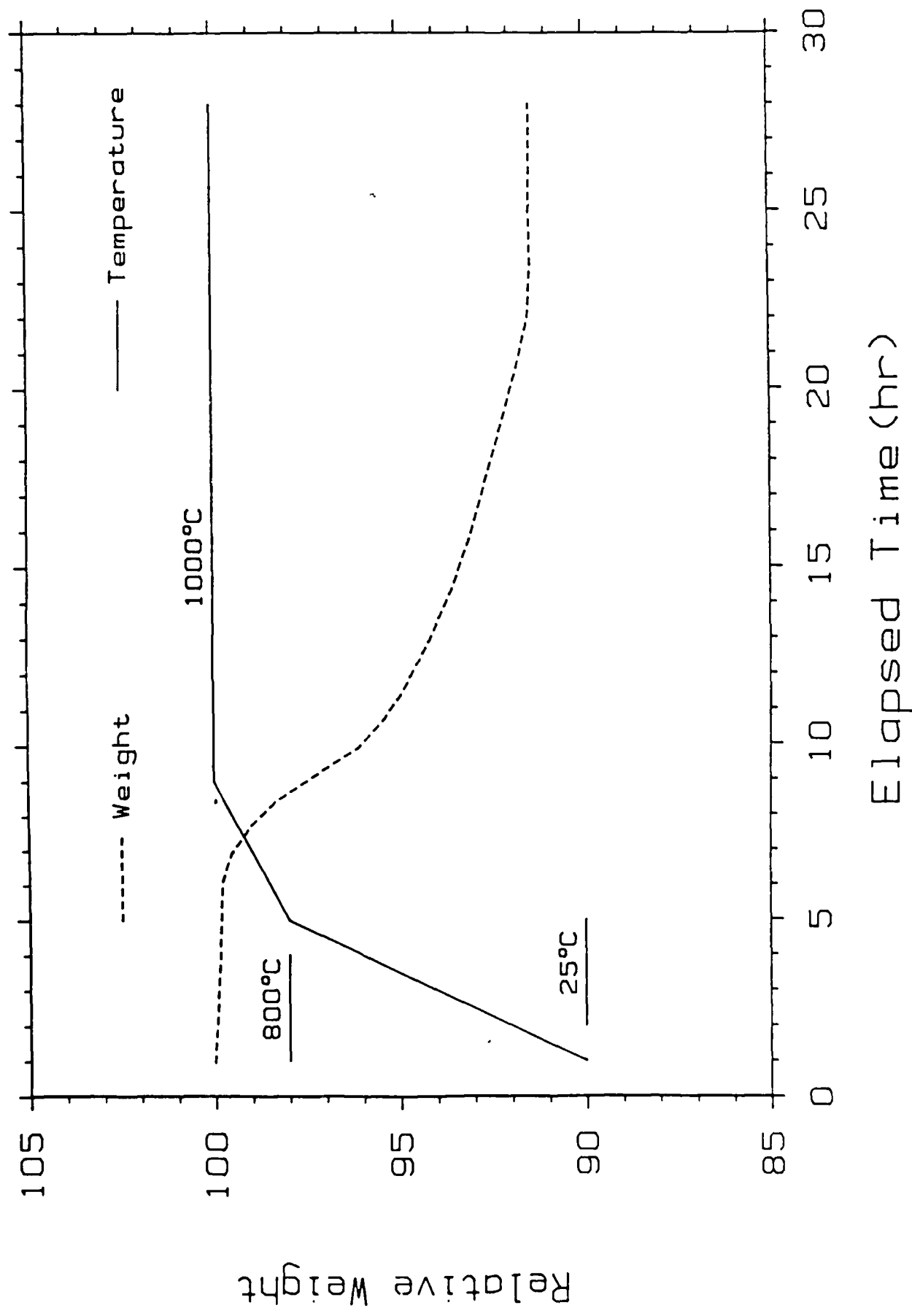


X-ray Patterns

Relative Intensity



TPR of Rh/Cr₂O₃ Mixture (Rh/Cr=2/1)



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